



Eco-friendly surfactant to demulsification water in oil emulsion: synthesis, characterization and application

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ABSTRACT

The need to replace of petroleum-based oil field chemicals with materials sourced from natural products has received significant attention owing to the eco-friendliness of these nature-based materials. In this study, we focused on the synthesis of a surfactant for use as a demulsifier for a water-in-oil emulsion. The demulsifier was synthesised via condensation reactions of corn oil with diethanolamine in the presence of a catalyst. The surfactant was characterised by FTIR, NMR, and GC-MS analyses. The separation efficiency of the surfactant was studied using bottle tests. The water separation from the emulsion 97% was achieved. The influence of the concentration and settling time on the demulsification efficiency was investigated. The result showed that increasing the concentration and settling time resulted in increased separation efficiency of the emulsion. The results of this investigation demonstrated the suitability of using corn oil in the synthesis of a surfactant as a demulsifier for crude oil emulsion. Nevertheless, more investigations are required to determine the mechanisms to optimise the system for industrial applications.

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Specifications Table

Subject area	Chemical Engineering, surfactant, oil and gas.
Compounds	diethanolamine, corn oil, p-Toluenesulfonic acid monohydrate, Petroleum ether and crude oil.
Data category	synthesized, physicochemical.
Data acquisition format	Elemental analysis by FTIR, NMR, and GC-MS.
Data type	Raw, separation, analyzed.
Procedure	Synthesis of surfactant to separation of water from crude oil emulsion by use of corn oil as raw material.
Data accessibility	Data are with the article in graphical presentations

1. Rationale

Water-in-oil (w/o) emulsions are formed during the production of crude oil, which is often accompanied by water. Nearly 90% of crude includes w/o emulsions, and a proportion of this is usually dispersed as small droplets throughout the crude oil depending on the production conditions [1,2]. Most processes involved in crude oil production are affected by the presence of water in crude oil; it affects the production, processing, and transportation processes, thus increasing the downtime in the sector [3,4]. Some of the problems associated with w/o emulsions include lack of space for processing and transport tasks, pipelines and production plant corrosion (aggravated by the dissolved salts present in the water phase), and reduced

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oil recovery. Moreover, w/o emulsions are highly viscous and cause catalyst poisoning and difficulty in oil transportation for downstream refinery operations [5]. The API gravity is also affected, and they cause environmental problems associated with the oil content in the wastewater [6].

To reduce the problems affined to crude oil emulsions and address environmental worries, the players in the petroleum sector must strive to avoid both the formation and breaking of w/o emulsions [7]. Demulsifiers are used as process aids during crude oil production to separate emulsified water from the produced oil [8]. Demulsifiers are surfactants that can destabilise w/o emulsions by promoting the aggregation and coalescence of water droplets and facilitating the water separation process from the oil [9]. Crude oil demulsification is a tedious and costly process in the oil industry; therefore, there is a need to use methods that are more effective [2,10]. The available crude oil demulsification methods are generally classified as electrical, thermal [11,12], mechanical [13], or chemical methods [14,15]. The most common and most effective demulsification method is chemical demulsification, which is a technically feasible way to treat w/o emulsions [16,17]. There are limitations to crude oil demulsification techniques owing to the high process cost, longer process time, and environmental situation [18,19]. Modern demulsifiers must ensure complete water and salt removal and must be cost-efficient, accessible, less toxic, and, above all, must consume fewer resources per ton of oil [20].

Numerous efforts are underway to develop low-cost chemicals from natural resources for successful application to chemically enhanced oil demulsification [21]. Thus, products synthesised from renewable raw material have received a considerable concern for their implementation to chemically enhanced oil recovery (EOR) [22,23], because of their excellent physicochemical properties and cheap for production [24]. lately, a lot of surfactants have been synthesised from vegetable oils [25–27] due to their renewable resource, universal availability, outstanding physicochemical property, efficient production and low costs, easy handling and environmentally friendly [28]. After the demulsification process, these demulsifiers can be easily separated and degraded to reduce its impact on the environment. Babu et al. synthesised anionic surfactants via sulfonation of ricinoleic acid methyl ester in castor oil. the results indicated that the synthesised surfactant decreases the interfacial tension IFT to a minimum value [29]. Negm et al. studied the prepare of biodegradable non-ionic surfactants from Jatropa oil [30]. other authors also informed the synthesis and characterisation of several surfactants from various vegetable oils, such as palm, mahua, and sunflower oil [31,32]. Vegetable oil is a considerable source of fatty acids, particularly base fatty acids [33]. Bio-enzyme catalysed esterification has been employed to synthesise fatty acid esters from vegetable oils, such as castor oil and Jatropa oil [29,34,35]. According to a previous study by Nurainia [36], the amine demulsifier group exhibited low toxicity and higher efficiency for breaking up an emulsion compared with alcohol, polyhydric alcohol. The disadvantage of these studies is that the use of many raw chemical materials leads to complex reactions that continue for several hours, thus increasing the processes time, energy consumption, and cost. They have also not been applied to the processes of breaking emulsions. Thus, it is important to use few raw materials to ensure a short reaction time during the preparation of a demulsifier. As well, Traditional surfactants are generally petroleum-based are not environmentally friendly. They can increase water toxicity when the separated water is discharged. which will result in higher processing cost [37].

Corn oil is an interesting oil crop because of its composition and favourable characteristics, and it is produced in big quantities in the world. Corn oil is a substantial ingredient of many foods and has a special taste [38]. Because of its effort-less digestibility, corn oil has additionally implementation in medicine. Moreover, newly, corn oil has been used in biodiesel manufacturing, mostly with ethanol production [39]. One can anticipate that the effect of corn oil chemical products is identical to products produced from other vegetable oils. The use of corn oil as a renewable source will lessen resource weather change because the greenhouse gas, CO₂, is consumed by plants through photosynthesis. Moreover, it will reduce the passive effect on air, water, land, and biodiversity and encourage agricultural development and rural economic growth [40]. This study investigated the applicability of corn oil for demulsifying w/o emulsions. The objective of this study was to synthesise a new demulsifier that is environmentally friendly and economical by using corn oil for application in the separation of w/o emulsions. The chemical structure of the demulsifier was confirmed by FTIR, NMR, and GC–MS.

2. Procedure

2.1. Materials

Reagent grade diethanolamine (>98.0%) and corn oil were supplied by R&M (Malaysia). p-Toluenesulfonic acid monohydrate (Reagent Plus®, ≥98%, solid) were purchased from Sigma-Aldrich (USA). Petroleum ether (40–60 °C b.p) was bought from Fisher Chemical (UK). The crude oil was collected from the Petronas Refinery in Melaka, and its components are shown in Table 1.

2.2. Synthesis of the demulsifier

First, 100 mL of the demulsifier was produced by reacting diethanolamine (33.33 mL) with corn oil (66.66 mL) in the existence of a standard catalyst. The corn oil and catalyst (p-toluene sulfonic acid, 0.83 g) were charged in a 250 mL laboratory reaction flask equipped with a reflux condenser, magnetic stirrer, and thermometer. The reaction mixture was gently heated to 120 °C with stirring until the catalyst dissolved and the diethanolamine was then slowly added. Subsequently, the temperature was increased gradually until it reached 175 °C within 1 h. The reaction was continued for 2 h under continuous stirring at a fixed temperature of 175 °C until the theoretical amount of water was collected. The product was obtained

Table 1
Physicochemical properties of the crude oil at room temperature.

Density	0.8628 (g/cm ³)
Viscosity	35 (N/m ²)
API Gravity	26
Surface Tension	24.678 (mN/m)
Interfacial Tension	14.731 (mN/m)

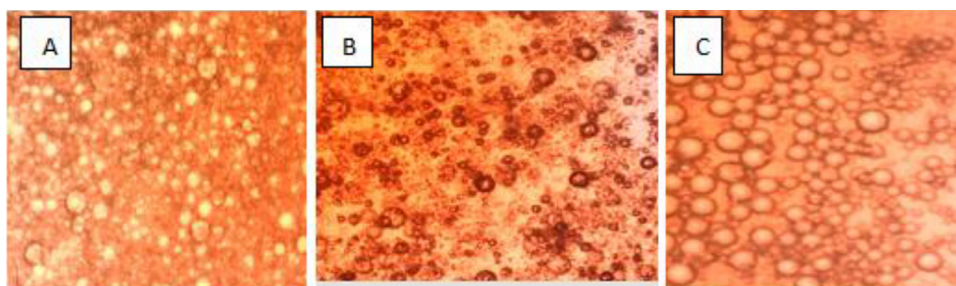


Fig. 1. Optical microscope photos of W/O (30/70 vol%) emulsion at different times a) 0, b) 10 and c) 20 min using 1.5% of ump surfactant.

after collecting 8 mL of water. Then, the sample was removed from the heating mantle and kept cool. The final product was purified and washed with petroleum ether and evaporated in a vacuum evaporator.

2.3. Emulsion preparation

For the chemical demulsification study, 300 ml of w/o emulsions (30/70 vol%) were prepared at ambient temperature. The preparation was performed using a standard 3-blade propeller revolving at 1500 rpm for 5 min. The water phase of the set-up was tap water (dispersed phase), and the oil phase was crude oil (continuous phase). The agent-in-oil technique was adopted in this study, i.e., the demulsifier was dissolved in oil, followed by gradual addition of water to the mixture. For emulsion formation and stabilisation during the study, the UMP surfactant (NS-16-1) was used. Fig. 1 shows the microscopic view of the emulsion and the development of small-sized homogenous emulsion droplets. This confirmed the stability of the emulsions.

3. Data, value and evaluation

3.1. Sample characterisation

The FTIR analysis was performed using a Nicolet iS5 FTIR spectrophotometer in the wavelength range of 4000–400 cm⁻¹ to identify the functional groups present in the synthesised demulsifier. The KBr method was used for this analysis. Identification of the chemical structure of the demulsifier was achieved using ¹H and ¹³C NMR spectroscopy (Model: 400 MHz Bruker Avance DRX-400 spectrometer, Germany). For NMR, CDCl₃ was used as the solvent. Gas chromatography (Model: Agilent 19091S-433) was employed for the determination of the components in the synthesised demulsifier from corn oil.

3.2. Fourier transform infrared spectroscopy (FTIR)

The FTIR spectrum of the demulsifier is depicted in Fig. 2. Here, the absorption peak at 3365 cm⁻¹ is due to the -NH bond stretching vibration (secondary amine), and the peak at 3007 cm⁻¹ is due to the presence of a =C-H bond. The peaks at 2922 cm⁻¹ and 2852 cm⁻¹ correspond to the asymmetric and symmetric stretching vibrations of -CH₂, respectively [41,42]. The peak at 1737 cm⁻¹ is attributed to the stretching vibration of the C=O bond (the main functional group of esters) [43]. The peak at 1458 cm⁻¹ is due to the bending vibration of -CH₂ and -CH₃ groups, and the peak at 1363 cm⁻¹ corresponds to the asymmetrical axial stretching of C-O in esters [44]. Additionally, peaks associated with C-N groups are at 1161 cm⁻¹ and 1047 cm⁻¹ [45] (Table 2).

3.3. Nuclear magnetic resonance spectroscopy (NMR)

The structure of the compound was further evaluated via ¹H nuclear magnetic resonance spectroscopy (NMR) and ¹³C NMR spectroscopy. The NMR peaks with their chemical shifts, multiplicities, coupling constants, and correlations were succinctly determined. The structure of the demulsifier is shown in Fig. 3a. Based on the ¹H NMR (CDCl₃, 600 MHz) spectrum of the

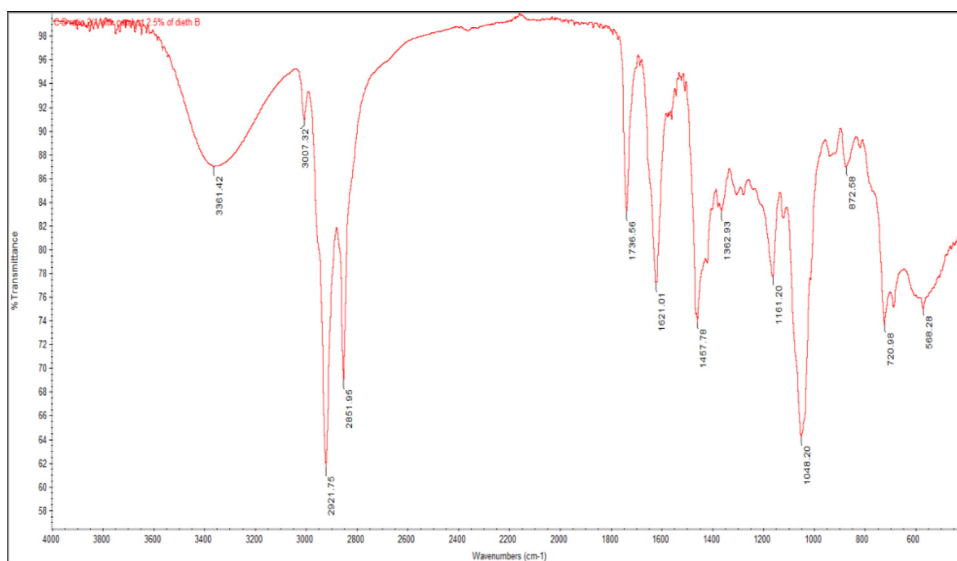
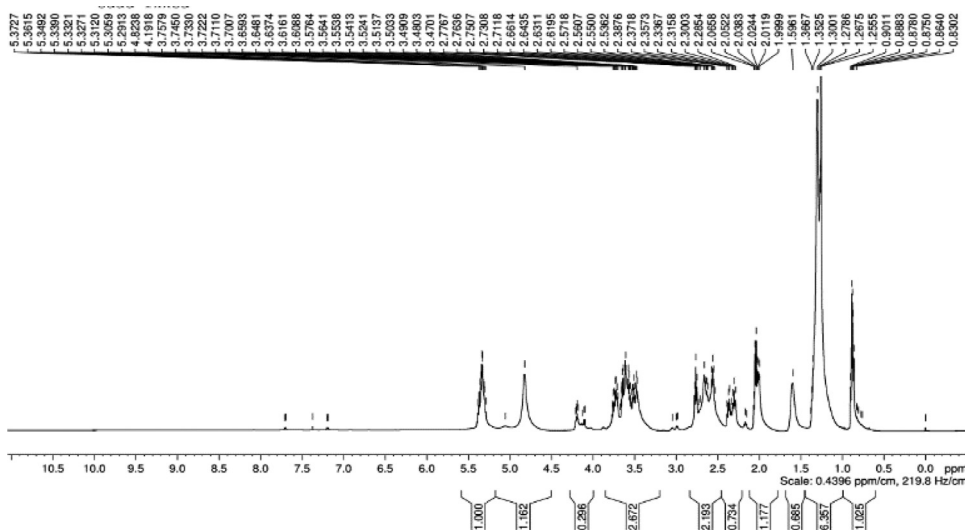


Fig. 2. FTIR spectrum of the demulsifier.

Table 2

Functional groups and wavenumber assignments.

Functional Group	wavenumber (cm ⁻¹)
C=C	1648
C=O	1622
C-N	1188
C-H	2926-2854
=CH and =CH ₂	3106-970

Fig. 3. ¹Hydrogen Nuclear Magnetic Resonance Spectroscopy (¹H-NMR).

chemical constituents, the double peak and single peak observed in the spectrum of the demulsifier at δ 0.8–1.6 ppm represents amine groups (NH₂ and NH), and the ¹H-NMR spectrum of the demulsifier showed a complicated aromatic proton multiplet at δ 2–4.3 ppm that was possibly due to the attachment of CH₂ groups directly to a hexahydro group [46]. Furthermore, there is an amino proton singlet at δ 4.3–5.4 ppm downfield from the usual aromatic proton region. The ¹³C-NMR spectrum of the demulsifier in Fig. 3b shows peaks at δ = 14.05–130.04 ppm that are possibly due to the respective attachment of C to the aromatic ring and amine groups [28].

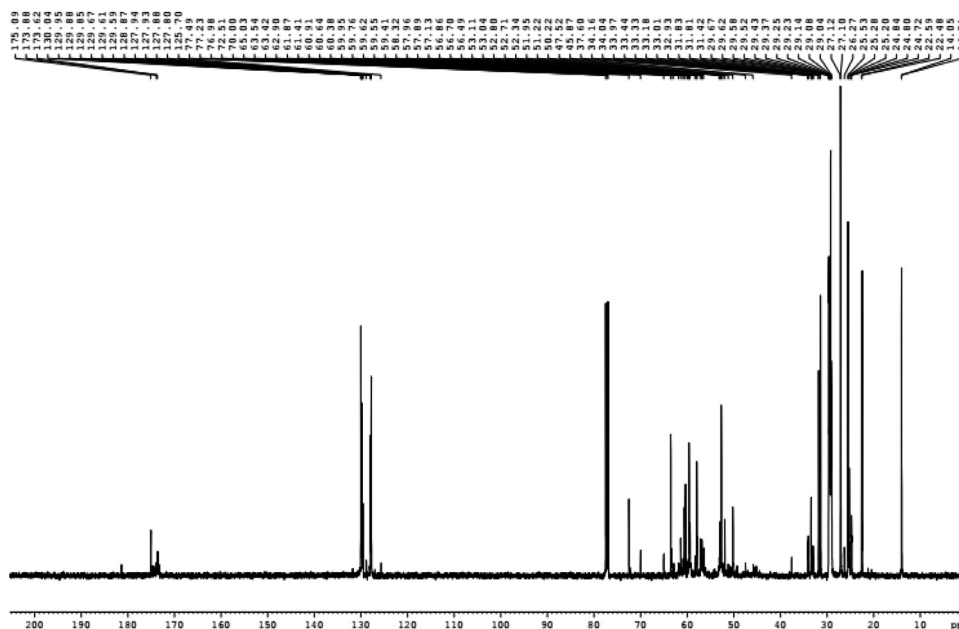


Fig. 4. ^{13}C Carbon Nuclear Magnetic Resonance Spectroscopy (^{13}C -NMR).

Table 3

GC-MS analysis results for the main compounds of the surfactant.

No	PK	RT	Area%	CAS#	Compound name	Molecular Formula
1	25	14.854	14.15	003443-84-3	9-Octadecenoic acid (Z)-	$\text{C}_{18}\text{H}_{34}\text{O}_2$
2	31	15.014	5.15	5906-35-4	1H-Azepin-1-amine, hexahydro-	$\text{C}_6\text{H}_{14}\text{N}_2$
3	33	15.613	4.47	000123-00-2	4-Morpholinepropanamine	$\text{C}_7\text{H}_{16}\text{N}_2\text{O}$
4	39	17.215	10	1000133-00-5	2-Amino-4-methyl-4-pentenoic acid	$\text{C}_6\text{H}_{11}\text{NO}_2$

3.4. Gas chromatography–mass spectroscopy analysis (GC–MS)

The demulsifier was subjected to gas chromatography–mass spectroscopy (GC–MS) analysis by injecting 1 μL of the sample in a gas chromatograph (Agilent 7890) coupled to a 5973 quadrupole mass spectrometer. The instrument was equipped with a HP-5MS capillary column (30 m \times 250 μm , film thickness = 0.25 μm). The operation conditions of the GC–MS analysis were set as follows: injection mode = splitless, injector temperature = 300 $^{\circ}\text{C}$, injector pressure = 7.6522 psi, transfer line = 316 $^{\circ}\text{C}$, oven temperature programmed as follows: 50 $^{\circ}\text{C}$ for 1 min, then 25 $^{\circ}\text{C min}^{-1}$ to 200 $^{\circ}\text{C}$, then 8 $^{\circ}\text{C min}^{-1}$ to 316 $^{\circ}$, carrier gas = helium at 1.5 mL.min^{-1} , and ion source temperature = 350 $^{\circ}\text{C}$ [47]. The compounds found included esters, amines, hydrocarbons, carboxyles, amides, and fatty acids (Table 3).

3.5. Effect of the settling time on the separation performance

The effectiveness of the prepared demulsifier in resolving w/o emulsions was evaluated using the graduated bottle test (Sany-glass). The concentrations of the demulsifier (2500 ppm) were added to the sample (50 mL) of the emulsion. The separation efficiency (SE%) of water was calculated from the observed volume of water in the beaker in mL using Eq. (1). The separation of phases was reported as a time function, as shown in Fig. 4. The results indicated that the separation of water increased with an increase in settling time; within 48 h, the separation efficiency approached nearly 95 %. In general, the graph indicates three rates of separation. Among 24 h the demulsification recorded the highest separation at a rate of 80%. A second region the Fig. shows a decline in separation rate, achieving 13% through the next 12 h. approximately 94% of the water has been separated from the emulsion during 36 h. At this instance, the presence of water droplets in the oil phase becomes minimal, hence, distancing the water droplets in the oil phase farther apart and reducing the possibility of water droplets colliding and coalescing. As a result, a much slower separation rate was observed through next time. As 94.5% water removal was achieved, no further separation was visibly observed as the settling time was extended until 72 hr. The results indicated that settling time is a significant factor affecting demulsification efficiency. This result elucidates

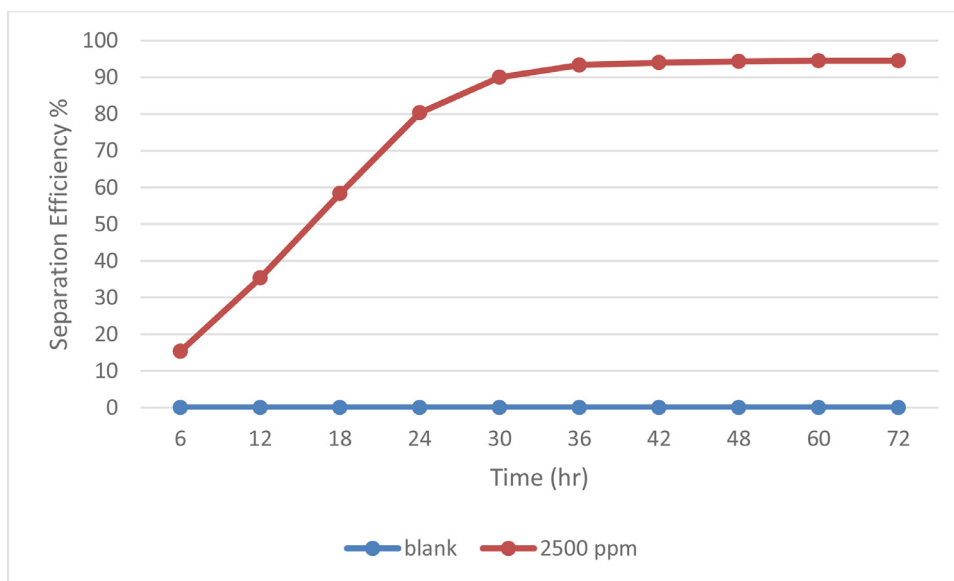


Fig. 5. Separation efficiency of the demulsifier as a function of time.

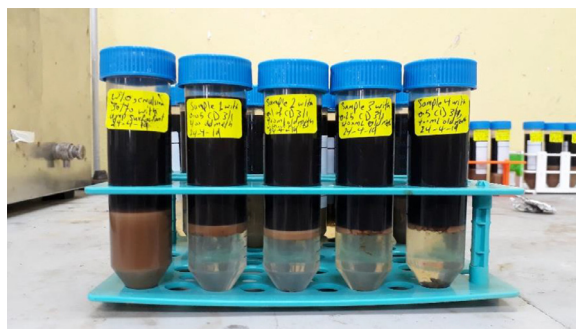


Fig. 6. Image of a demulsification water-in-oil emulsion.

that the solution time is an important factor that influences the demulsification performance [43].

$$\text{separation efficiency \%} = \frac{\text{Volume of separated water in mL}}{\text{Original volume of water in the emulsion, mL}} \times 100 \quad (1)$$

3.6. Effect of concentration on the separation performance

The aim of this test was to determine the relationship between the concentration of the demulsifier and the water separation efficiency. Different concentrations of the demulsifier were used for this test at room temperature in a similar experimental procedure as described above. The concentrations used for this test were 1000, 2000, 3000, and 4000 ppm. The volume of separated water from the emulsion system was recorded, as shown in Fig. 5, and the difference in the water removal ratio versus the demulsifier concentration for different demulsifiers are shown in Fig. 6.

The data confirm that water separated at $\geq 93\%$ for all the used concentrations. However, the blank samples could not separate any water droplet for more than one month. At 1000 ppm, the water separation was 90%, whereas the 2000-ppm concentration achieved approximately 93.33%. At 3000 and 4000 ppm, the water separation was 95 and 96.66% respectively during 36 hr. The highest separation was observed 93, 95, 96, and 97% at 1000, 2000, 3000, 4000 ppm of demulsifier respectively among 60 hr. No more separation followed after this time. The results indicated that the separation efficiency ratio consistently increased with increasing demulsifier concentration. Hence, a high concentration can significantly accelerate the demulsification progress. Therefore, the demulsifier concentration is a critical parameter in demulsification processes [48].

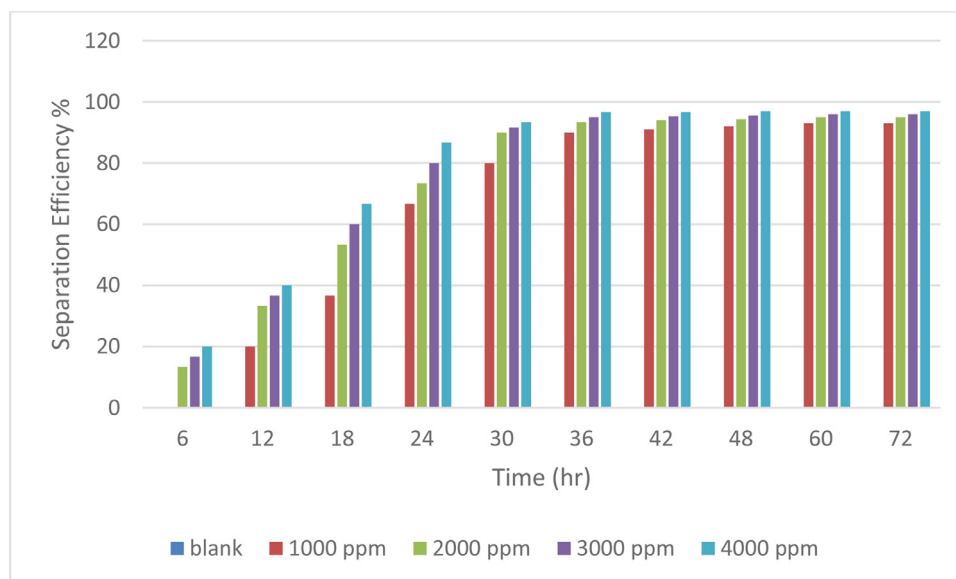


Fig. 7. separation efficiency % at different concentration.

4. Conclusions

The suitability of our synthesised demulsifier from corn oil was successfully tested for w/o emulsion demulsification. The demulsifier was characterised using NMR, FTIR, and GC-MS techniques to ascertain the functional groups present in the surfactant. The demulsifier demonstrated excellent performance; the separation efficiency ratio of 97% was achieved. The efficiency of emulsion separation increases with increasing settling time and the doses of surfactant. The samples showed a difference in the percentage of water separation as follows 93%, 95%, 96%, and 97% by injection of 1000, 2000, 3000, and 4000 ppm of the demulsifier, respectively.

CRediT authorship contribution statement

• All authors have participated in (a) conception and design, or analysis and interpretation of the data; (b) drafting the article or revising it critically for important intellectual content; and (c) approval of the final version. • This manuscript has not been submitted to, nor is under review at, another journal or other publishing venue. • The following authors have affiliations with organizations with direct or indirect financial interest in the subject matter discussed in the manuscript: M. A. Saad, N. H. Abdurahman, Rosli Mohd Yunus, Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang.

Declaration of Competing Interest

None.

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